



**National
Safety
Council**

Data Sheet
1 655 Rev 87

Recognition and Handling of Peroxidizable Compounds

PEROXIDE FORMATION in solvents and reagents has caused many accidents. Every worker must learn to recognize and safely handle peroxidizable compounds. Peroxides form by the reaction of a peroxidizable compound with molecular oxygen through a process called autoxidation or peroxidation. Peroxidizable compounds are insidious. Under normal storage conditions they can form and accumulate peroxides which may explode violently when subject to thermal or mechanical shock.

2 Peroxides in solution at concentrations up to about 1 percent do not normally present thermal or shock hazards. Such solutions may be safely disposed of or treated to remove peroxides. However, should crystals form in a peroxidizable liquid or discoloration occur in a peroxidizable solid, peroxidation may have occurred and the product should be considered extremely dangerous and destroyed without opening the container.

3 To prevent accidents caused by peroxidizable compounds, users of these substances should develop a program for their recognition and safe handling. This program should emphasize:

- Recognition of chemical structures that may form peroxides
- Use of warning labels
- Controlled inventory of peroxidizable compounds
- Use of peroxide detection tests and peroxide removal procedures

Proper safety equipment and process procedures

4 This data sheet will cover recognition of peroxidizable compounds, storage and handling of such compounds, and tests for determining the amount of peroxide that has formed in a sample of a peroxidizable compound. The safety practices outlined in this data sheet should reasonably assure safe use of peroxidizable compounds in the laboratory.

Recognition

Peroxidizable chemical structures

5 Presence of one of the peroxidizable structures listed in Table I in a compound is a warning that peroxides can form and a hazard may exist. Recognition of these structures is also important because products resulting from peroxidation are impurities that may alter chemical processes using the parent compound.

6 The degree of danger varies considerably with structure. Peroxidation of organic structures 1 to 11 in Table I has caused many explosions. Structures 12 to 14 have caused only a few and none have been reported for structure 15. Peroxidation of inorganic structures, namely alkali metals, alkoxides, and amides, also have resulted in many explosions.

7 The readiness with which any of the peroxidation structures in Table I form peroxide is highly dependent on the bonded (attached) chemical

groups. For example, most ethers that have an alkyl group bonded to the oxygen atom of structure 1 will readily peroxidize and present a significant hazard. In contrast, ethers having an aromatic group bonded to the oxygen atom of structure 1 generally do not peroxidize under normal conditions and can be handled without the precautions needed for peroxidizable compounds.

8 The hazard associated with a given peroxidizable structure generally decreases for its higher molecular weight derivatives. For example, the peroxide of butyl ether has less explosive energy potential than that of ethyl ether. Ten or more carbon atoms at a peroxidizable site usually are low risk systems.

Peroxidizable compounds

9 Common compounds that form peroxides during storage are in Lists A, B, and C of Table II. The time period for testing the opened container is indicated in parenthesis. *These lists are representative rather than exhaustive.*

10 The most hazardous compounds—those that form peroxides which may explode even without being concentrated—are in List A. Compounds forming peroxides that are hazardous only on concentration, such as distillation or evaporation, form List B. List C is made up of vinyl monomers that may form peroxides that can initiate explosive

PEROXIDE FORMING STRUCTURES

Organic structures (in pp) in order of decreasing hazard

Formula 1		Formula 8	
1 	Ethers with hydroperoxide groups (ethyl hydroperoxide)	9 	Alkyl ethers that contain tertiary hydrogen atom (isopropyl benzene)
Formula 2		Formula 10	
2 	Alcohols with alpha hydrogens (tert-butyl alcohol)	10 	Alkanes and cycloalkanes that contain tertiary hydrogen atoms (ethylcyclohexane)
Formula 3		Formula 11	
3 	Olefins with allylic hydrogen atoms (butylene cyclohexane)	11 	Acrylates and methacrylates (methyl methacrylate, acrylonitrile)
Formula 4		Formula 12	
4 	Chlorides (chloroform, chloroacetylene)	12 	Secondary alcohols (see butyl alcohol, diphenylmethanol)
Formula 5		Formula 13	
5 	Vinyl chloride and vinylidene chloride (vinyl chloride)	13 	Aldehydes (benzaldehyde)
Formula 6		Formula 14	
6 	Dienes (butadiene, chloroprene)	14 	Ketones with alpha hydrogen atoms (diisopropyl ketone, MEK)
Formula 7		Formula 15	
7 	Vinyl acetate with alpha hydrogens (diacetylene vinyl acetate)	15 	Ureas, amides, lactams with hydrogen atom on carbon atom attached to nitrogen (N-ethylacetamide, N-isopropylacetamide)
Formula 8			
8 	Alkyl isobutylene with hydroperoxide (3-methylbut-3-ene)		

Inorganic Substances

1 Alkali metals, especially potassium, rubidium and cesium

2 Metal amides (sodamide)

Organometallic Substances

1 Compounds having a carbonyl bond (methylmagnesium iodide, dimethylmagnesium chloride, butyllithium)

2 Metal alkoxides (potassium tert-butoxide, sodium propoxide)

polymerization of the bulk monomers

11 Peroxide accumulation is a balance between the rate of peroxide formation and the rate of peroxide degradation for the particular substance under the environment of the sample. For example, certain highly reactive compounds such as organometallics accumulate peroxide at low temperatures because the peroxide degradation rate is slowed relative to the formation rate. In contrast, less reactive compounds such as hydrocarbons or ethers are usually best kept at low temperatures.

12 The more volatile the peroxidizable compound, the easier it is to concentrate the peroxides. One should also remember that pure compounds are more subject to peroxide accumulation because impurities may inhibit peroxide formation or catalyze their slow decomposition.

Detection of peroxides

13 Always suspect peroxidizable compounds of containing peroxides. For organic liquids (solvents), use the test procedures for detecting peroxides described below. No suitable simple test procedure is available for detection of peroxides in substances such as alkali metals, alkali metal alkoxides or amides, or organometallics. The test procedures described below should not be used with these metal-containing peroxidizable compounds. The test procedures are also not suitable for gases, since any peroxide formed from a gas will be less volatile than the gas itself.

14 Testing peroxidizable solvents for peroxides prior to distillation should be routine. A little practice with standard peroxide detection procedures enables a chemist to make a rough estimate of the quantity of peroxide. Some peroxide tests, which from a safety viewpoint are satisfactory for monitoring laboratory samples, are the iodide tests, the ferrous thiocyanate test, and a simple test strip.

15 The Iodide Test is based on the oxidation of iodide to iodine by the peroxide. Several procedures have been employed as follows:

- **Method A**—Add 0.5–1.0 ml of

Common Compounds That Form Peroxides During Storage

List A—Red Label (Three Months) Peroxide Hazard on Storage	List B—Yellow Label (Twelve Months) Peroxide Hazard on Concentration	List C—Yellow Label (Twelve Months) Hazard Due to Peroxide Initiation of Polymerization
Isopropyl ether Divinyl acetylene Vinylidene chloride Potassium metal Sodium amide	Ethyl ether Tetrahydrofuran Dioxane Acetal Methyl t-butyl keton Ethylene glycol dimethyl ether (glyme) Vinyl ethers Dicyclopentadiene Diacytylene Methyl acetylene Cumene Tetrahydronaphthalene Cyclohexene Methylcyclopentane t-Butyl alcohol	Styrene Butadiene Tetrafluoroethylene Chlorotrifluoroethylene Vinyl acetylene Vinyl acetate Vinyl chloride Vinyl pyridine Chlorobutadiene (Chloroprene) 9,10-Dihydroanthracene Indene Dibenzocyclopentadiene

*When stored as liquid, the peroxide-forming potential increases and certain of these monomers (especially butadiene, chloroprene, and tetrafluoroethylene) should then be considered as List A compounds.

the material to be tested to an equal volume of glacial acetic acid to which has been added about 0.1 g of sodium iodide or potassium iodide crystals. A yellow color indicates a low concentration of peroxide in the sample; a brown color indicates a high concentration. A blank determination should be made. Always prepare the iodide/acetic acid mixture at the time the test is made, because air oxidation slowly turns the blank to a brown color.

Method B—This procedure calls for addition of 1 ml of a freshly prepared 10 percent aqueous solution of potassium iodide to 10 ml of the organic liquid in a 25 ml glass stoppered cylinder of colorless glass that is protected from strong light. A yellow color (brown at high peroxide content) indicates the presence of peroxide. For this test, the color formed and the peroxide content have the semi-quantitative relationships shown in Table III.

16 **Method A** is preferred. In studies summarized in Table IV, color development by Method A was rapid, whereas up to 10 minutes were required for color formation by Method B. Note that the color intensity for a given peroxide content may be influenced by the nature of the medium.

17 The Ferrous Thiocyanate Test is a very sensitive test based on the reaction of peroxides with colorless ferrothiocyanate to form red ferrothiocyanate. In this test, a drop of the compound is mixed with a drop of sodium ferrothiocyanate reagent. The latter is prepared by dissolving 9 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in 50 ml of 18 percent hydrochloric acid. Add granulated zinc (0.5 to 1.0 g) followed by 5 g of sodium thiocyanate when the transient red color fades; add 12 g more of sodium thiocyanate and decant the liquid from the unused zinc into a clean stoppered bottle.

18 Limited test data¹ indicate the relationship between color and peroxide content shown in Table V.

19 Remember that these tests are valid only for relatively simple chemicals. Some organic compounds may also act as oxidizing agents and therefore appear to give positive tests for peroxides. When dealing with peroxidizable materials that may be insoluble in the test solution, it may be necessary to use a co-solvent such as peroxide-free isopropanol to facilitate the peroxide test.

20 **Peroxide Test Strips** are commercially available for qualitative and semi-quantitative testing. These

convenient to use however they do not have the universality or the sensitivity of the iodide test and have limited shelf life

21 When used to detect peroxides in readily volatile ether the test strip is immersed in the ether withdrawn and then breathed upon As the vaporizing ether absorbs heat moisture in the breath condenses to form a reproducible water film in which the detection reaction takes place The dipping and breathing can be repeated to improve sensitivity to less than 1 ppm for diethyl ether peroxide for example

22 With aqueous solutions it is not necessary to breathe on the strip Simply immerse the strip wait the prescribed time and compare with the color scale supplied by the manufacturer

23 The test involves a two step conversion of oxygen in the peroxy group (including hydrogen peroxide sodium peroxide sodium perborate as well as organic peroxides found in diethyl ether tetrahydrofuran and dioxane) to a blue oxidation product

Storage and Handling Procedures

Inventory control

24 Each person responsible for a laboratory should be required to make and maintain an inventory of the peroxidizable materials in the laboratory Review the inventory every three months either testing for peroxide or discarding all List A (Table II) samples three months or older and Lists B and C (Table II) samples 12 months or older

25 Quantities of peroxidizable compounds should be purchased according to short term needs to assure that peroxide buildup which may accompany long term storage is minimized Record the date of receipt on the label to assist in first in—first out usage of materials (see paragraphs 26 27) Purchasing of package sizes corresponding to use requirements will also minimize exposure to air from multiple openings of the container

Labeling

26 All materials in Lists A B and

peroxidizable compound should have a warning label bearing the date of receipt in the laboratory and the date when the container was first opened Storeroom items should have the label affixed by storeroom personnel For non storeroom items or for materials synthesized in the laboratory the individual worker should attach the warning label

27 A 1½ x 3 in label is recommended For compounds in List A (Table II) or any other peroxidizable compound that represents a hazard on storage white or yellow lettering on a red background is suggested This label should read DISCARD OR TEST WITHIN THREE MONTHS AFTER OPENING For materials in Lists B and C (Table II) or other similar peroxidizable materials use a yellow label with red lettering This label should read DISCARD OR TEST WITHIN 12 MONTHS AFTER OPENING (see Figure 1) These storage periods are recommended for items stored under the conditions and precautions described below

Storage

28 All peroxidizable compounds should be stored away from heat and light Sunlight is an especially strong promoter of peroxidation Protection from physical damage and ignition sources during storage is also essential

29 The recommendations that follow are for ordinary storage conditions When storage conditions are unusual special precautions must be taken For example at tropical temperatures inspection of the material should be made more frequently because leakage around closures becomes more prevalent which increases the chance of formation of explosive peroxides at the closures

30 Most common container materials such as steel stainless steel aluminum nickel copper baked phenolic linings and ceramics are suitable for containers in which oxidizable compounds are stored All metal containers however must be clean and free from metal oxides because some metal oxides such as iron or copper oxide may actually promote peroxide formation

31 Particular care should be given

containers Loose or leaky closures may permit evaporation of storage material leaving a hazardous concentration of peroxides in the container When handling peroxidizable compounds stored in cylinders care must be taken to ensure that the cylinders are maintained free from air

32 Peroxidizable compounds should be stored under an inert (oxygen free) atmosphere such as nitrogen Vinyl monomers containing certain inhibitors are exceptions (see paragraph 36) Containers should be purged with nitrogen if opened and closed during storage and handling

33 The use of oxidation inhibitors is especially important in the safe handling of peroxidizable materials Hydroquinone alkyl phenols aromatic amines or similar materials are recommended by the manufacturers as being effective in preventing peroxide formation during storage of peroxidizable compounds The inhibitor selected should be compatible with use or purity requirements of the compound A program of periodic testing and replenishing inhibitor levels should be followed during storage of

PEROXIDIZABLE COMPOUND

RECEIVED OPENED

DATE

DISCARD OR TEST WITHIN
3 MONTHS AFTER OPENING

PEROXIDIZABLE COMPOUND

RECEIVED OPENED

DATE

DISCARD OR TEST WITHIN
12 MONTHS AFTER OPENING

Figure 1 Two labels differing in color and message are recommended for use with peroxidizable compounds that represent a storage hazard For List A compounds (see Table II) the suggested label (shown at top) should have white or yellow lettering on a red background and the message should read Discard or test within three months after opening For materials in Lists B and C (Table II) the suggested label should be yellow with red lettering and it should read Discard or test within 12 months after opening

TABLE III
The Iodide Test for Peroxide (Method B)

Color Reaction	P	C	nt P	id P	I	H O
Barely discernible yellow				0.001 to 0.005		
Definite yellow color				0.01		
Brown color				?		

A percentage of 0.01 more hazardous

peroxidizable material ⁴⁴

34 List A (Table II) materials—that can accumulate a hazardous level of peroxides simply on storage after exposure to air—should be evaluated for peroxide content at least every three months after opening and either re dated if safe or else treated or discarded. Before disposal of any List A material, review the properties of the material (if possible in consultation with the organization's safety professional) to assure safe disposal.

35 List B (Table II) materials should not be stored for longer than 12 months after opening unless a suitable test shows they have not accumulated peroxide. If List B material gives a significantly positive test (medium to deep yellow by iodide test or red by the ferrous thiocyanate test) but must be retained, it must be treated to remove peroxide, repackaged, shown by test to be free of peroxide, and re dated on its label.

36 List C (Table II) materials are vinyl monomers and should be stored for no longer than 12 months unless test results show them to be free of peroxide. Commercial vinyl monomers usually contain additives (inhibitors) which inhibit peroxidation. Generally, storage of inhibited vinyl monomers should be under air rather than nitrogen or other inert atmosphere because customary inhibitors are phenolic compounds which require oxygen for their action. Most vinyl monomers may be polymerized without removal of inhibitor by proper adjustment of initiator concentration, thus making the isolation of the more hazardous uninhibited material unnecessary.

37 Uninhibited List C materials can be a significant hazard. Quantities of such uninhibited monomers greater than 500 g should be stored for no longer than 24 hours. Small samples (less than 10 g) may be stored longer than 24 hours only with discretion. Generally, storage

of uninhibited vinyl monomers should be under nitrogen and below room temperatures. For storage in excess of 24 hours, a suitable inhibitor should be added, and its name and quantity should be placed on the label.

38 Methyl methacrylate, ethyl acrylate, and other common acrylic monomers are *not* in List C because there has been no report of their peroxidation to hazardous levels in normal use and storage. Peroxidizable compound warning labels and other peroxidizable compound safety procedures are not required for the acrylic monomers. However, for good safety practice, acrylic monomers should be inhibited for storage, and work with uninhibited monomers should be limited to quantities less than 500 g.

37 With the approval of the organization's safety professional or safety committee, certain solid or high boiling organic peroxidizable compounds may be exempt from the storage rules. Such compounds are those that, by the nature of their use, will not be concentrated, e.g., surfactants or plasticizers containing polyether groups. These compounds

should have a boiling point above 300 C (572 F) or a vapor pressure below 0.1 mm Hg (338 Pa) at 20 C (68 F) so that there is not opportunity for concentration. Even though such compounds are not believed to represent a significant hazard, the impurities introduced as a result of peroxidation can alter chemical processes using the material.

Removal of peroxides

40 There are several established laboratory procedures for removing peroxides from solvents. To ensure that peroxide removal has been accomplished, the treated solvent should always be re tested for the presence of peroxide.

41 Method 1: Peroxides can be conveniently removed by passing the solvent through a short column of ordinary activated alumina. No water is introduced, and small amounts of water that may be present are removed. This method is effective for both water insoluble solvents and water soluble solvents (except for lower alcohols). When traces of peroxide cannot be tolerated, solvent flow can be directed into the reaction vessel from the alumina column. Effectiveness of Method 1 was determined by Dasler and Bauer.

42 Care should be exercised in disposing of the activated alumina after this operation because certain peroxides remain unchanged on the alumina. Further, such a high surface material treated with a

TABLE IV
THE IODIDE TEST FOR PEROXIDE
Method A vs. Method B (Reaction Time and Color Development for Various Compounds is Affected by Both the Compound and the Per Cent of Peroxide)

Compound	Per Cent Peroxide as H ₂ O	Test Method	
		Method A	Method B
Ethyl ether	0.010	Medium yellow—Fast	Pale yellow—Slow to develop
Tetrahydrofuran	0.0047	Medium yellow—Fast	Pale yellow—30 to 60 seconds
Tetrahydrofuran	0.0082	Deep yellow	Deep yellow
Dioxane	0.0155	Deep yellow	Deep yellow to brown

The iodide test will differ with the basic compound peroxide percentage is changed

hazard and therefore should not be disposed of in a waste can that contains other flammable materials. It is recommended that alumina used for peroxide removal be flushed with or drowned in a dilute acid solution of potassium iodide or ferrous sulfate.

43 Method 2 Peroxide impurities in water insoluble solvents (ether hydrocarbons etc) are easily removed by shaking with a concentrated solution of a ferrous salt. A frequently used ferrous salt solution can be prepared either from 60 g or ferrous sulfate 6 ml of concentrated sulfuric acid and 110 ml of water or from 100 g of ferrous sulfate 42 ml of concentrated hydrochloric acid and 85 ml of water (With some ethers traces of aldehydes are produced by this treatment) Water is introduced by this method so post drying will be required if a dry solvent is wanted.

44 Other Methods Among the many other methods reported for peroxide removal are treatment with amines aqueous sodium metabisulfite⁴ and stannous chloride sodium hydroxide and cerous hydroxide. Some active metals and their derivatives—sodium lithium aluminum hydride carbon hydride and sodium borohydride—may be used to destroy small quantities of peroxides⁵. However do not use them with halogen containing solvents or active hydrogen compounds (alcohols phenols etc). Peroxides have also been removed from ethers by

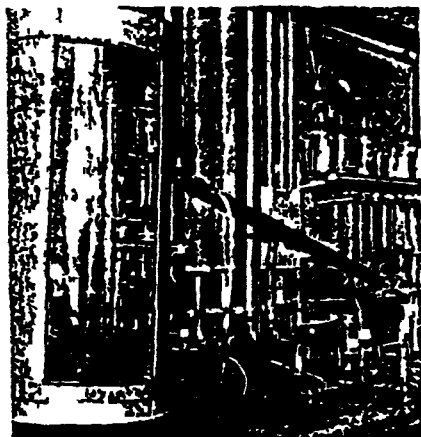


Figure 2 To avoid accidents associated with distillation of peroxidizable compounds when they have been concentrated in the distillation residue add a high-molecular weight inerting solvent that will not distill such as mineral oil or phthalate ester.

TABLE V
THE FERROUS THIOCYANATE TEST
Relation Between Color Change and Peroxide Content of a Compound

Color	Per Cent of Peroxide as H ₂ O
Barely discernible pink	0.001
Pink to cherry red	0.002
Red	0.008
Deep red	0.04

A percentage of 0.008 or more is a hazard.

percolation through a column of ion exchange resin.

Disposal

45 Compounds that are suspected of having very high peroxide levels because of visual observation of unusual viscosity or crystal formation or because of age should be considered extremely dangerous. The precautions taken for disposal of these materials should be the same as for any material that can be detonated by friction or shock.

46 In general the material should be carefully removed using explosive handling procedures from the laboratory to a remote area where it can be safely destroyed preferably by burning. *It is of the utmost importance that the container not be opened.* The act of opening the container could detonate peroxide crystals around the container cap or other closure.

47 If adequate personal protective equipment and technical expertise for handling potentially explosive substances are not available at the laboratory site then a professional disposal company or local police or other bomb disposal authority should be contacted for assistance.

Distillation and evaporation precautions

48 Test for peroxides before distilling or evaporating any List A or B (Table II) material. If positive (medium to deep yellow by iodide test or red by the ferrous thiocyanate test) discard the material or remove the peroxides. Before distilling any List C (Table II) material a suitable

polymerization inhibitor should be added.

49 Most accidents associated with distillation of peroxidizable compounds have occurred when peroxides have become concentrated in the distillation residue. Therefore addition of a high molecular weight inerting solvent which will not distill such as mineral oil or a phthalate ester is recommended (see Figure 2). The inerting solvent acts as a desensitizing diluent for residual peroxides when distillation is completed. Should such a diluent be undesirable and the material is shown by test to have no more than a trace of peroxide distillation leaving at least a 10 per cent heel is a suitable alternative procedure. Never distill to a dry residue.

50 A shield should be used when evaporating or distilling mixtures that may contain peroxidizable compounds. Safety glasses and a face shield are essential at all times.

51 A boiling aid or a magnetic stirrer should be used in preference to a nitrogen bleed to maintain ebullition. If a bleed is the only effective arrangement be doubly sure that an inert gas is used—never air.

52 In higher boiling peroxidizable compounds such as alkyl ethers or di- or triethylene glycol the peroxides formed are ordinarily decomposed thermally by the heat required for distillation at atmospheric pressure. However when such higher boiling peroxidizable compounds are distilled at a reduced pressure the boiling temperature may be lower than the peroxide decomposition temperature and concentration to a hazardous explosive mixture can result.

Safety audit

53 Before starting any chemical process whether in the laboratory or in the manufacturing area perform a safety audit including a review of possible hazards due to peroxidizable compounds. Peroxidation may have already occurred in one or more of the starting materials; it may occur during the process or in the storage of the products. In every chemical process the following factors which were discussed in this data sheet should be considered relative to (a) the starting materials (b) the process itself and (c) the products

- *Structure*—are peroxidizable structures present or being formed?

- *Process conditions*—will the process condition favor initiation of peroxidation and accumulation of peroxides?

Storage—will storage containers and conditioners reduce peroxide initiation and accumulation and are all products properly inhibited and labeled?

54 Should the audit indicate that peroxidation or peroxide presence is likely the described procedures of

handling, testing and removal should be followed. As noted previously, peroxidation in a chemical process may not only be a serious hazard because of the explosion potential but it also may affect the results of the process through lower yield and unwanted impurities.

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